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Final Report on

STUDY AND DESIGN OF MULTIPLE OR STACKED DETECTORS AND A SINGLE FIXED DETECTOR

Prepared for

MANNED SPACECRAFT CENTER
NATIONAL AERONAUTICS & SPACE ADMINISTRATION
HOUSTON, TEXAS

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AMERICAN MACHINE & FOUNDRY COMPANY
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I. INTRODUCTION

For the analysis of lunar samples by x-ray diffraction, an instrument is required which is capable of performing a measurement within a reasonably short period of time. Many instrumental and operational problems may arise if the measurement itself requires a long interval of time. Thus, a diffractometer which simultaneously detects and records the essential portion of the diffracted x-ray spectrum would be vastly superior to a design utilizing a mechanical scanning mechanism which moves a detector through the diffracted beam. The superiority of the simultaneously detected and recorded technique, which is fundamentally an x-ray camera design, is attributable to: (1) the conservation of electrical energy required to power the scanning mechanism; (2) reduction of the total time required to obtain the analytical data; (3) elimination of the possibility of garbled data due to variations in the intensity of the primary x-ray beam resulting from vacillation in the primary power source; and (4) elimination of moving mechanisms and other minor mechanical and operational considerations.

In the typical x-ray diffractometer a sample is placed in a collimated beam of monochromatic x-radiation and the incident radiation is diffracted at specific 2θ angles in accordance with Bragg's law. A conventional diffractometer has a single detector which integrates the diffracted radiation as the detector moves through a particular arc of 2θ angles. Scanning time is variable on the normal laboratory instrument, so that some trade-off may be made between the time required to obtain an x-ray pattern and the characteristics of the individual reflection (such as intensity, spread, resolution, etc.). Typical operating times for a scanning goniometer are around 90 minutes per pattern. With simultaneous detection and recording this time can be substantially reduced. Evidence shows that integration times of six minutes or less are very plausible.

The purpose of this program was to establish feasibility and design concepts utilizing multiple or stacked detectors, a single fixed detector, or other pertinent detectors which appear suitable to detect and record an x-ray diffraction spectrum without utilizing mechanical scanning mechanisms.

Because the nature of the program was inherently continuous, many of the phases overlapped. They were, however, divisible into broad categories: namely, optics, detectors, and data handling and presentation.

II. X-RAY OPTICS

A comprehensive study of optic systems of various x-ray camera arrangements was made during the initial part of the program. Aisde from the more obvious factors which apply specifically to the camera (e.g., intensity of the incident beam, theoretical resolution, size and weight), it was necessary to consider also compatibility with various detector designs.

Our analysis indicated only marginal acceptability of the Debye-Scherrer camera design. The advantages of this type of camera lie mainly in the fact that the resulting data cover almost 180° 2θ with a single exposure plus the fact that a double set of diffraction lines are simultaneously recorded for each exposure. This is advantageous since it would permit utilization of a greater number of detectors. The principal disadvantages are: (1) the diffracted intensities are very low owing to the small cross-section of the incident beam; (2) sample minuteness; (3) the problem of sample preparation and loading; and (4) high precision alignment requirements after the specimen is loaded into the camera.

The feasibility of the Debye-Scherrer camera design was determined by measuring the combined intensities of the 100 and 101 reflections from a powdered sample of α-quartz. This was accomplished by placing a scintillation counter, from a standard x-ray diffractometer, beside a Debye-Scherrer powder camera which had previously been positioned and aligned according to normal x-ray camera practice. The experimental arrangement is shown in Figure 1. By positioning the scintillation counter in the above-described manner, that portion of the two diffracted beams which would normally fall on the camera cover was available for measurement. Using CuKα radiation, at a power level of approximately 950 watts, the diffracted intensities of the 100 and 101 reflections were only around 30 c/s. Direct beam intensity was approximately 92, 200 c/s. The ratio of the direct to diffracted intensity (3×10^3 c/s) was considered to be sufficient evidence to indicate this procedure would, at best, be marginal.

Based on the above considerations, this camera design was deemed less satisfactory than an arrangement using focusing optics.

The back-reflection type of x-ray camera is also unsuitable for use as a lunar diffractometer. In a back-reflection camera pattern, only those reflections are present which lie in the back-reflection quadrant. Inasmuch as the principal identifying lines of the majority of known compounds lie in the forward-reflection region, this optic arrangement was likewise adjudged less desirable.

By utilizing focusing geometry, cameras can be constructed which give much greater resolution and intensity without increased exposure times as compared with Debye-Scherrer cameras of the same radius. Cameras with this optical arrangement are frequently called Seemann-Bohlin cameras. Furthermore, specimen preparation and loading are much simpler than with Debye-Scherrer cameras.

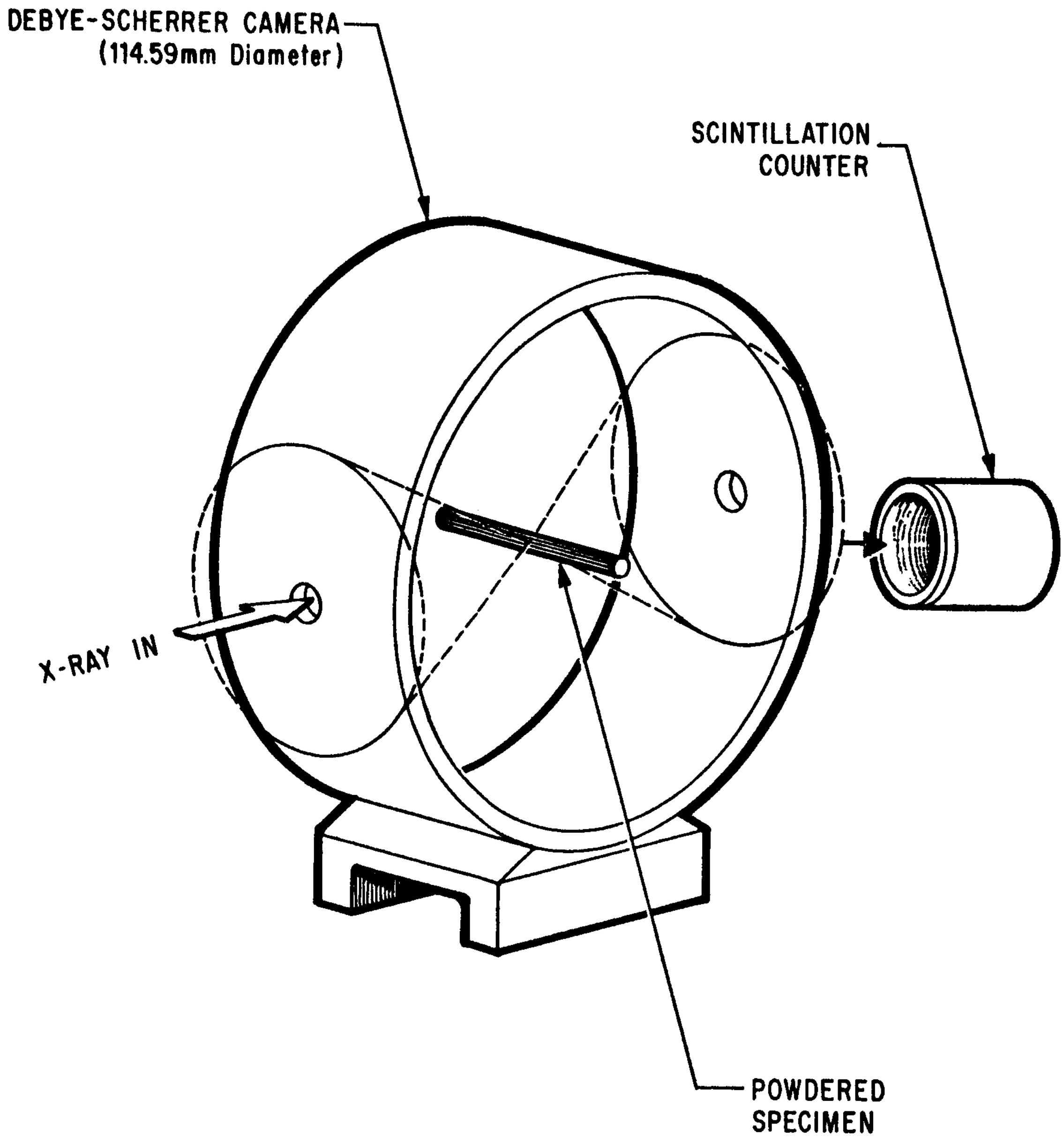


Figure 1. Experimental Arrangement to Determine Intensity of the 100 and 101 Reflections of Alpha Quartz.

The superiority of curved optics versus a flat optic system is shown in Table 1. These data were obtained by scanning a conventional goniometer through the 200 reflection of aluminum using normal laboratory diffraction procedures. Curved and flat samples were used. As seen in Table I, in all cases the intensity (measured as peak intensity above background) of the curved sample is greater than for the flat sample. On the average, curved optics will increase the intensity of a diffracted beam by a factor of 16 to one. Other desirable considerations are also shown in Table 1. A 5° take-off angle provides distinct improvement in peak intensity when compared to a 1.5° take-off angle; secondly, and logically, the larger the divergence slit the greater the intensity. However, the divergence slit width is limited by other features within the system, namely, sample size. To capitalize on these advantages a focusing camera arrangement was constructed. Figure 2 is a schematic representation and Figure is a photograph of the actual mechanical arrangement. The camera was built around an available standard x-ray tube for the sake of convenience, although further substantial improvements may be realized by going to a smaller size x-ray tube. However, the particular dimensions chosen result in a radius of the Rowland circle which is very close to the radius of the focusing circle for the Surveyor Bragg-type diffractometer at the 101 diffracted line of quartz, therebyallowing a direct comparison of all performance characteristics of the two systems. The specific design parameters of the prototype camera are: (1) Rowland circle radius of either 8.8

TABLE I

COMPARISON OF CURVED VS. FLAT OPTIC SYSTEMS

DIVERGENCE SLIT	CURVED OPTICS (Relative Intensity, counts/sec)	FLAT OPTICS (Relative Intensity, counts/sec)
1.5° Take-off Angle		
1°	12	0.5
4°	59	3.5
No Slit	75	4.0
5° Take-off Angle		
1°	15	2.0
4°	88	5.5
No Slit	95	6.0

OPERATIONAL DATA:

CuKα radiation.
200 reflection of aluminum.
Radius of curved sample - 9.78".
Norelco Diffractometer with goniometer.
Divergence slits adjacent to x-ray source.
Take-off angle measured as the vertical drop from the shadow of the x-ray anode.

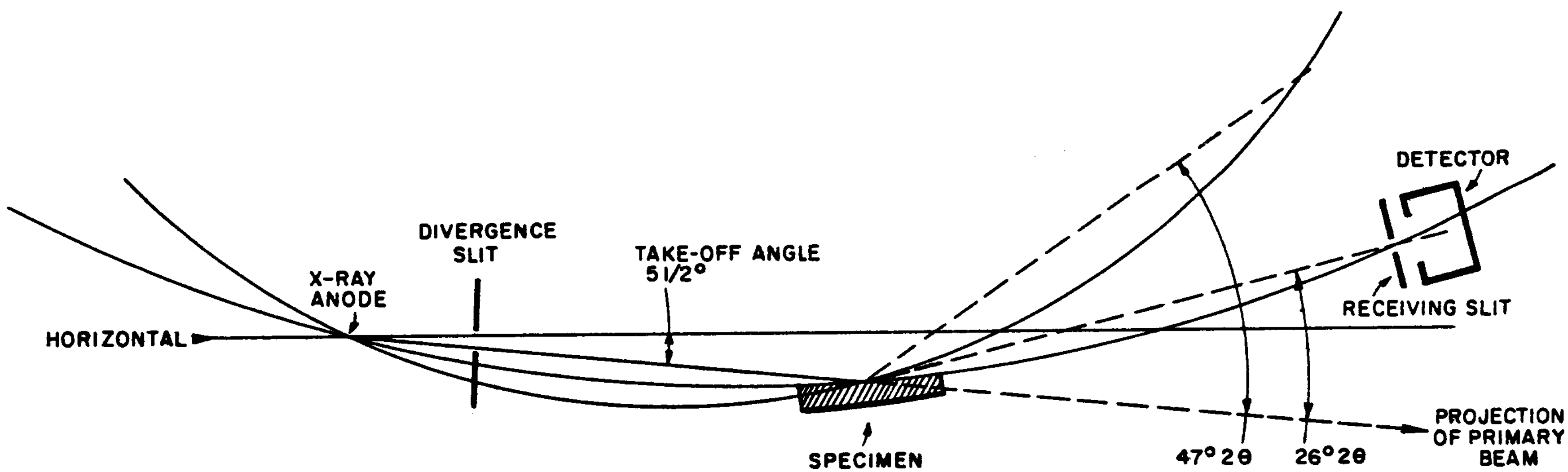


Figure 2. Focusing Geometry Schematic Diagram.

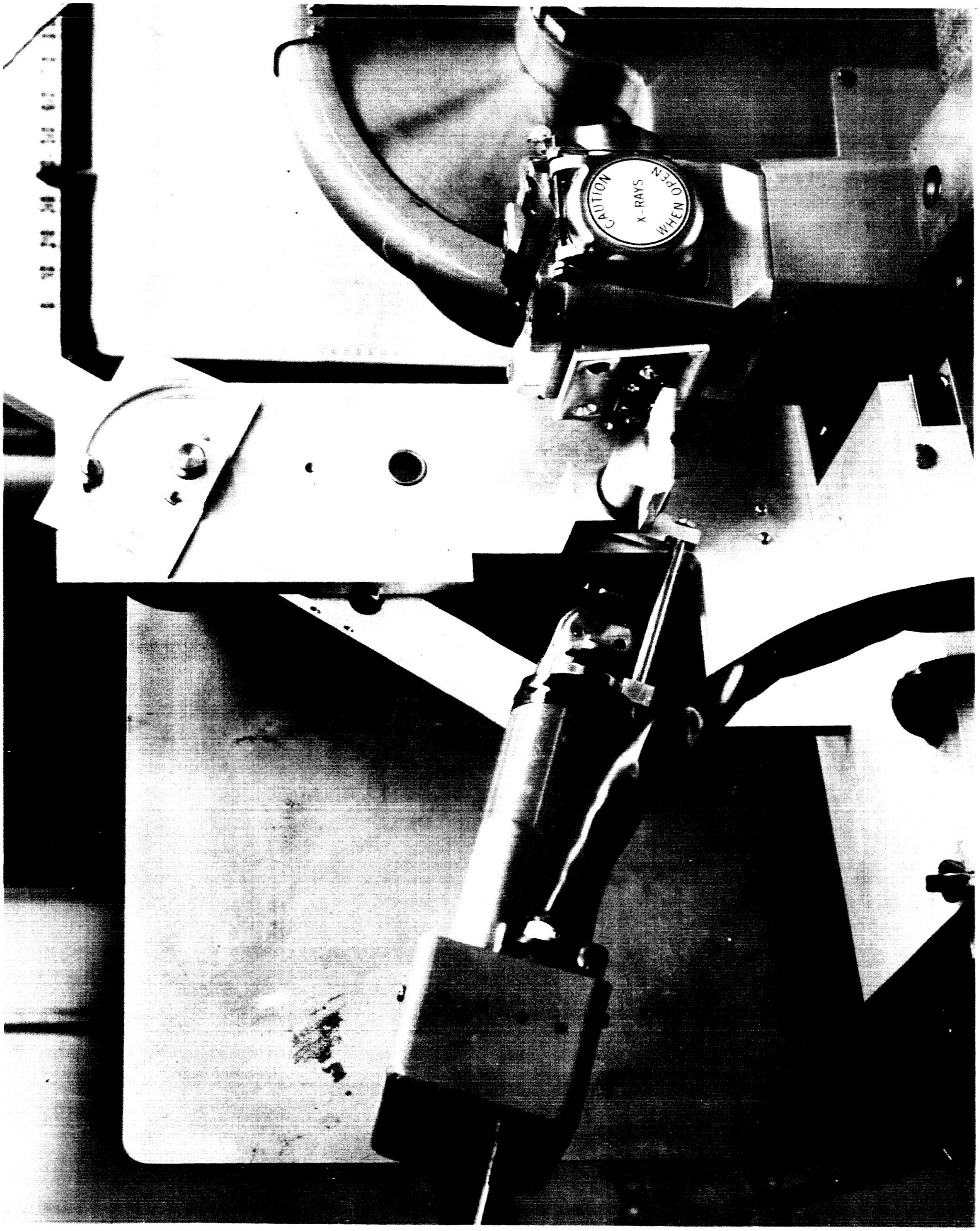


Figure 3 Focussing Camera with Scintillation Counter.

or 17.3 centimeters; (2) curved sample holders one and 1.5 inches long, respectively; (3) x-ray tube take-off angle of 5.5°; (4) anode-to-sample distance of 7.0 centimeters; (5) divergence slit width of 3mm and 3/8" long, and (6) focal spot to divergent slit distance of 4.6 cm. When the scintillation counter was used, an 0.006" receiving slit and a set of Soller slits were positioned adjacent to the detector. No Soller slits were incorporated in the divergence slit mechanism adjacent to the x-ray anode. A typical pattern of the first eight reflections (20.9° to 50.1° 2θ) from a sample of alpha quartz is shown in Figure 4. Figure 5 shows a typical pattern (36.6° to 77.6° 2θ). The patterns were made with different Rowland circles; hence the need for two patterns. The operational data for these patterns are summarized in Table II.

The main results obtained from these experimants are the peak intensities for the diffracted lines, the resolution of the lines, and the peak-to-background ratio. A peak intensity for the 101 reflection (Figure4) is on the order of 10,000 counts per second or about twice the value obtained on the Surveyor Bragg-type diffractometer under equivalent conditions. The resolution is 0.3° 2θ, which is somewhat worse than the Surveyor instrument, but it is probable that a more precise design and alignment would result in improved resolution. The most encouraging result, however, was the relatively high signal-to-background ratio obtained. The value of this ratio was about fifty, which is not worse

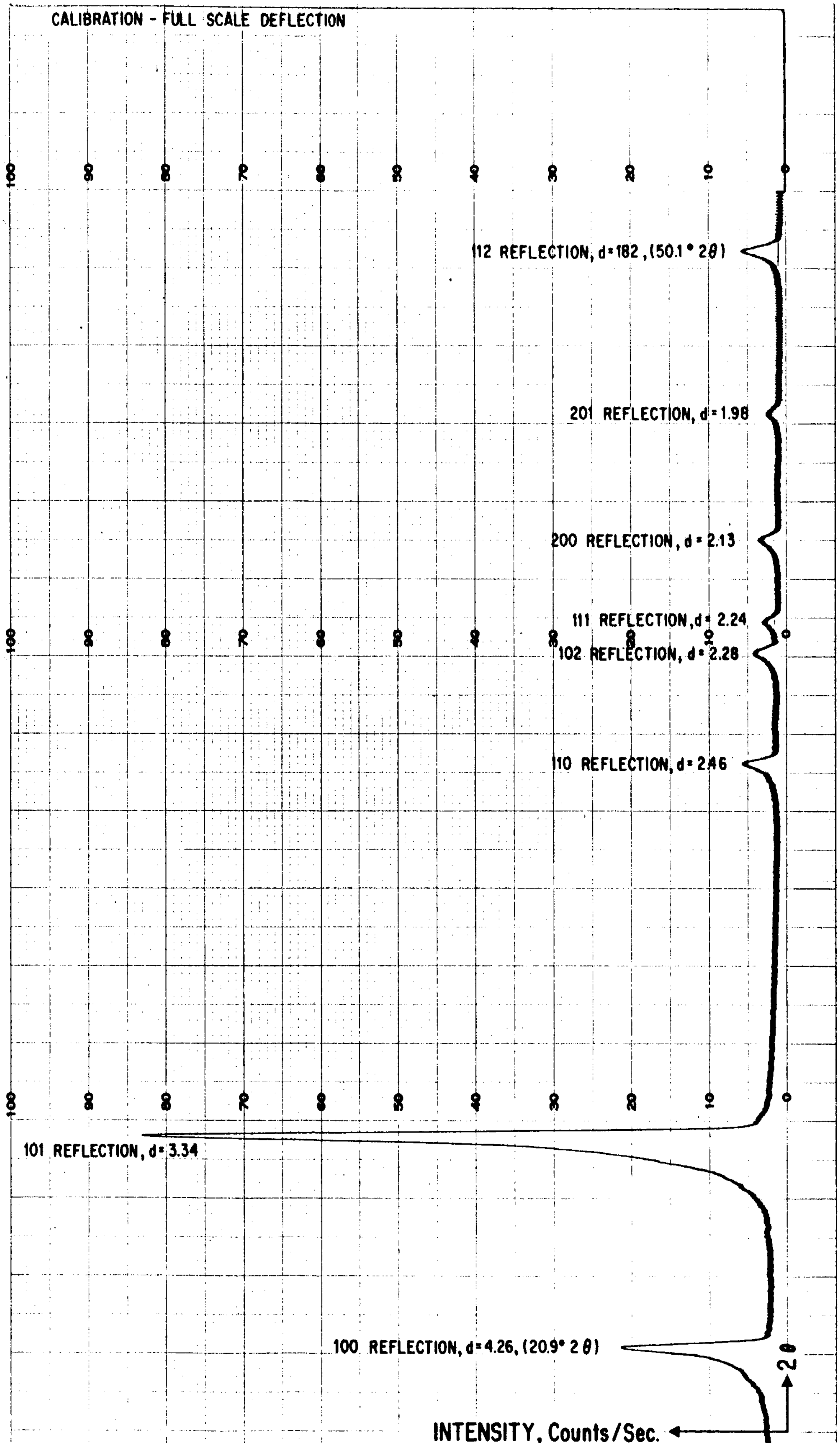


Figure 4. X-Ray Diffraction Pattern of the First Eight Reflections of Alpha Quartz.

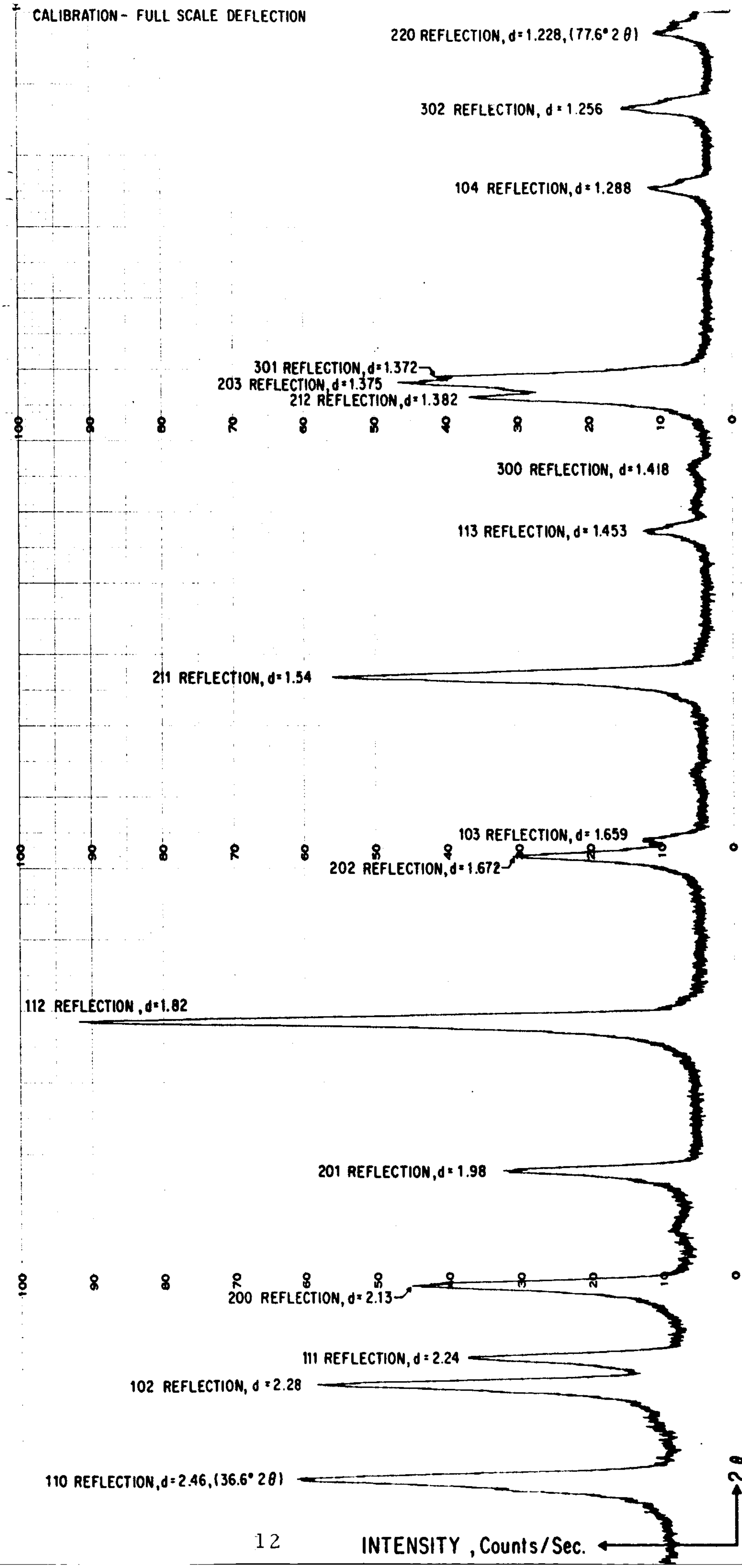


Figure 4. X-Ray Diffraction Pattern of Alpha Quartz.

TABLE II

OPERATIONAL DATA FOR TYPICAL X-RAY PATTERNS OF ALPHA QUARTZ

	Figure 4	Figure 5
Rowland Circle Radius	17.3 cm	8.8 cm
X-Ray Optics	Focusing Asymmetric	Focusing Asymmetric
Radiation	CuKα	CuKα
Power	25 watts	25 watts
Scale Factor	32	2
Multiplier	1	1
Time Constant	2	2
Pulse Height Discriminator:		
Scale Factor	8	8
Base Voltage	7.5	7.2
Window Voltage	18	18
Gain	20	20
Scintillation Counter Voltage	1000	1000
Divergence Slit	3 mm	3 mm
Receiving Slit	0.006"	0.003"
Scatter Slit	None	None
Full-Scale Deflection	12,800 c/s	800 c/s
Particle Size (Quartz)	-325	-325

than the Bragg-type diffractomer and fairly constant over a wide angular range.

For purposes of comparison, the maximum intensity of the strongest peak (112 reflection) in Figure 5 is on the order of 700 counts per second; resolution is $0.3^{\circ} 2\theta$; and peak-to-background ratio is about 15 to one.

Theoretical considerations indicate that intensity and signal-to-background ratio should improve with a smaller Rowland circle radius. This may be verified by comparing the 112 reflection in Figures 4 and 5. In Figure 4 (obtained with the larger Rowland circle) this reflection has an intensity of approximately 600 counts per second and peak-to-background ratio of five to one. In Figure 5 (obtained with the smaller circle) the peak intensity is approximatey 700 counts per second and peak-to-background ratio is 15 to one. Thus a threefold gain in peak-to-background ratio has been achieved. The reduction in the Rowland circle radius was not a complete scale-down inasmuch as the specimen-to-anode distance remained the same in both systems (see Figure 2). Thus, the change in the peak intensity of the 112 reflection is not as great as would be realized if the specimen-to-anode distance had been reduced proportionalely.

In Table III the absolute and relative peak intensities for all reflection planes from both Rowland circle patterns are summarized

TABLE III

COMPARISON OF EXPERIMENTAL PEAK
INTENSITIES WITH REFERENCE DATA (ALPHA QUARTZ)

Reflection Plane (hkl)	Figure 4 Intensity (chart division*) Back-Ground	Figure 4 Peak Above Back-Ground	Figure 4 Relative	Figure 4 Resolution ($^o2\theta$) (at half-height)	Figure 5 Intensity (chart division*) Back-ground	Figure 5 Peak Above Back-ground	Figure 5 Relative	Figure 5 Resolution ($^o2\theta$) at half-height)	ASTM Reference No. 5-0490 Interplanar Spacing(A)	ASTM Reference No. 5-0490 Relative Intensity c/s
100	3	18	22	0.3	-	-	-	-	4.26	35
101	2	81	100	0.3	-	-	-	-	3.34	100
110	2	4	5	0.3	10	52	10	0.4	2.46	12
102	1	3	4	0.4	7	51	10	0.4	2.28	12
111	1	2	2	0.3	7	30	6	0.3	2.24	6
200	1	3	4	0.4	7	38	8	0.3	2.13	9
201	1	2	2	0.4	6	26	5	0.3	1.98	6
112	1	5	6	0.3	6	86	17	0.3	1.82	17
202					4	27	5	0.3	1.67	7
103					4	8	2	-	1.66	3
211					4	52	10	0.3	1.54	15
113					4	8	2	0.4	1.45	3
300					4	2	<1	0.5	1.42	<1
212					4	33	6	-	1.382	7
203					4	43	9	-	1.375	11
301					4	37	7	-	1.372	9
104					3	8	2	0.4	1.288	3
302					3	11	2	0.5	1.256	4
220					3	8	2	0.5	1.228	2

*For absolute intensities see Table II

and compared with highly reliable reference data. The relative intensities of the experimental data were obtained by taking the absolute intensity of the specific reflection which lies nearest the design point of symmetry of each Rowland circle and setting this value equal to the intensity of the specific reflection as given by the ASTM. The design point of symmetry is the 2θ angle which lies on the Rowland circle at a distance from the center line of the sample which is equal to the distance from the center line of the sample and the x-ray anode (see Figure 2). For the Rowland circle with the 17.3 cm radius, the symmetry point is $26^{o}2\theta$, or approximately the 101 reflection; for the circle with the 8.8 cm radius the symmetry point is $47^{o}2\theta$, or approximately the 112 reflection. Analysis of Table III will indicate good agreement between the experimantal intensities and ASTM data, which for alpha quartz is considered to be highly reliable.

The asymmetric shape of the diffraction peaks, especially in Figure 4, is worthy of note. Instrumental factors are known which modify the profile and position of diffraction maxima.[1] These effects are generally manifested by peak broadening, shifting in position from the theoretical 2θ angle, and by rendering the peak asymmetric. It is most likely

1. Klug, H.P. and Alexander, L.E., X-Ray Diffraction Procedures, John Wiley, New York p 243.

that the symmetry of the peaks in Figure 4 is due to vertical divergence and absorption.[2]. Vertical divergence effects are relatively minor at Bragg angles around 90°, but with decreasing 2θ angle become more pronounced. This may be observed by comparing the diffraction peak from the 100 reflection of Figure 4 with the 211 reflection of Figure 5. Peak asymmetry due to absorption of the beam by the sample is ordinarily small and considered to be less significant but not excludable without investigation. The prototype camera arrangement designed and constructed to show the feasibility of a rapidly operating x-ray diffraction system contained no Soller-slit collimation (for vertical divergence) near the x-ray anode. In further modification of this camera design, the additional use of Soller slits near the detector are recommended, although in analyzing Figures 4 and 5 no detrimental effects from peak asymmetry were observed while making the analyses, aside from the askew appearance of the peaks. The use of Soller slits near the x-ray source will also assist in reducing the background with subsequent enhancement of the peak-to-background ratio.

2. Alexander, J., J. Appl. Phys., 25, 1954, p. 155-161.

III. DETECTOR SYSTEMS

In order to sense the presence of an x-ray spectrum it is necessary to use some sort of a detector. A good detector should have high efficiency, high output, good linearity, the required resolution, and discrimination against undesired background radiation. High efficiency is desirable to reduce the exposure time of the detector to the x-rays. High output is desirable to eliminate the need for signal amplifiers, thereby minimizing the quantity of associated electronics. Good linearity is necessary to count accurately the wide range of count rates encountered in the spectrum. The resolution must be good enough to read the positions of the spectrum peaks with sufficient accuracy to distinguish one material from another. The detector should be sensitive to the characteristic x-ray radiation but not to general x-ray radiation or cosmic radiation. For multiple detector applications it should have this discrimination without the aid of electronic circuit discrimination to keep the electronics to a minimum.

The following detectors were considered in this preliminary study phase: Individual stacked geiger counters, multiple geiger counters, scintillation counter, channel-multiplier-type counter, and x-ray vidicon. These counters were evaluated on the basis of their spatial resolution, efficiency, and sensitivity as well as the requirements with respect to power supply voltage, complexity of associated electronics, ruggedness, vacuum requirements, price, and temperature characteristics. Findings are summarized in Table IV. The following comments can be made.

TABLE IV

DETECTOR COMPARISONS

Characteristic	GM Single	GM Multiple	Scintillator	Channel Multiplier	X-Ray Vidicon
Resolution	Poor	Fair	Poor	Good	Very Good
Efficiency	Fair	Fair	High	Poor	Fair
Sensitivity	Good	Good	Fair	Fair	Fair
Counter Voltage	400	400	1000	3500	300
Readout	Complex	Medium Complex	Complex	Simple	Simple
Vacuum	No	No	No	Yes	No
Price	High	Medium	Medium	High	Medium
Ruggedness	Good	Good	Fair	Good	Good
Temperature	Good	Good	Good	Good	45°Cmax.

Individual stacked geiger counters have a problem of poor resolution. This problem, however, can be circumvented by stacking the counters into multiple rows. In order to achieve the required spatial resolution, a multiple array with about 500 individual counters, each corresponding to the minimum resolvable element, has to be realized. The complexity of such a system makes this approach less attractive than some of the other methods.

The multiple geiger counter consisting of many closely spaced, insulated sections has some attractive features. It was decided to use a geiger counter in preference to a proportional counter. The geiger counter does not require a high gain electronic amplifier. Considering that 500 individual channels are required, this will result in a substantial saving of electronics. As the estimated dead-time of the geiger counter is on the order of 50 microseconds, no coincidence losses would occur at the radiation intensity levels to be expected. Geiger tubes filled with argon or neon have an inherent discrimination against general radiation and cosmic radiation.

The scintillation counter, using fiber optic coupling to an image intensifier and this in turn coupled through fiber optics to an image orthicon tube, was studied but its application remains doubtful. The evaluation showed that the problem of optically coupling the scintillator to the fiber optics would result in poor spatial resolution. Furthermore, the extremely low light levels available require the use of a fairly complex image intensifier system. A possible solution to these problems might be the use of a new secondary emission, high sensitivity vidicon presently being developed for the Apollo program for a different application. This device, however, is not yet available.

The channel-multiplier-type counter was evaluated experimentally. This counter has desirable features except that it does require a somewhat high power supply voltage of 3500 volts. The main unknown, however, with this counter was its efficiency of x-ray detection. This parameter was measured and it was determined that the quantum efficiency is only 2.7 percent. While this value does not rule out the method completely, this and the fact the counter must be operated in vacuum make it appear disadvantageous.

The x-ray vidicon is a device equivalent to a regular vidicon except that it will convert x-rays directly into an electrical signal. This detector has excellent resolution but the output is at a low level, thus requiring a high gain amplifier.

On the basis of this evaluation, we decided to proceed with parallel approaches: one using a geiger counter and the other an x-ray vidicon.

A. X-RAY VIDICON

Preliminary work was done with a borrowed closed-circuit TV camera and monitor utilizing an x-ray sensitive vidicon tube in the camera.

The reflections from the 200 plane of a single crystal of lithium fluoride were observed on the TV monitor screen with an x-ray source equivalent to one having a 25-watt power input. Resolution of the diffracted beam was such that it was easy to discern $K\alpha_1$ and $K\alpha_2$.

It was reasoned that the sensitivity of the camera could be improved by a higher gain amplifier with decreased bandwidth to decrease amplifier noise. Integration of the signal would also be helpful to reduce the noise output. Slower sweeps would be necessary with the narrow band amplifier. The slower sweep or a wait period would also be desirable to allow the vidicon screen to build up a charge from low intensity radiation.

A camera was purchased and modified to provide sweeps with respect to an x-ray spectral line, as shown in Figure 6. A one-kilocycle

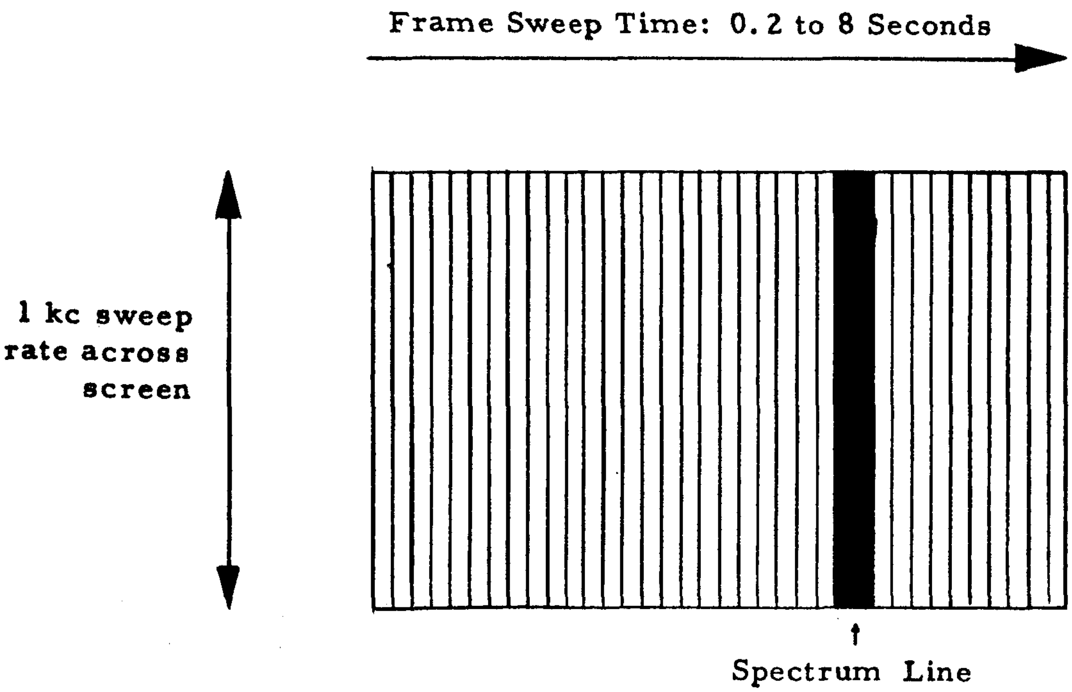


Figure 6. Sweep Signals on Vidicon Screen.

sweep across the screen was selected as slow enough to keep the amplifier bandwidth requirements low but high enough to eliminate the need for bulky coupling capacitors. With a frame sweep time of one second, the frame would contain 500 lines. An adjustable delay was provided between sweeps to allow the screen time to charge. This delay could be set from 0.5 to 30 seconds.

The output of this system was displayed on a standard oscilloscope with the scope sweep triggered to be concurrent with the camera frame sweep. The output of the amplifier representing a spectral line was integrated and applied to the vertical amplifier of the scope giving a presentation as shown in Figure 7.

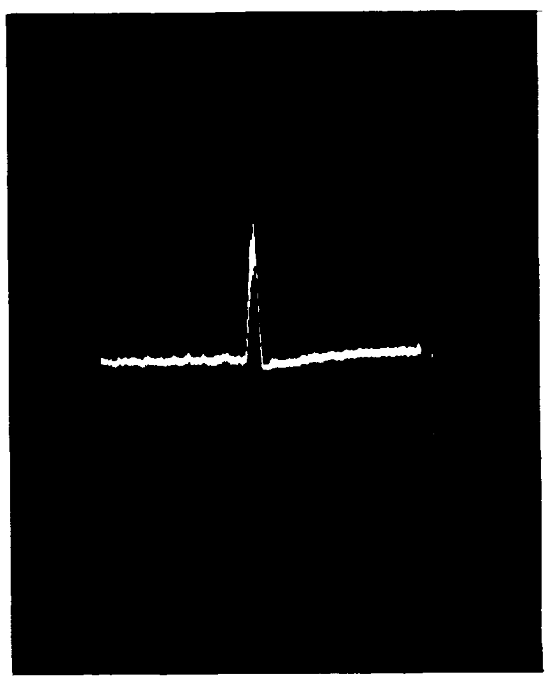


Figure 7. Oscilloscope Presentation.

The TV camera was set up in a calibrated direct beam from the x-ray generator with an 0.003 inch wide slit between the source and the vidicon screen. Nickel foils with known attenuation factors were inserted between the slit and the vidicon screen to vary the count rate. Figure 8 is a photograph of the vidicon assembly mounted in position for acceptance of a collimated x-ray beam.

The first work was done with two used x-ray vidicon tubes, Machlett type ML-589. The best sensitivity obtained with these tubes was 59,500 counts/sec/mm^2 with a signal-to-noise ratio of two to one. The screen was allowed to charge for 30 seconds and then read out with an 0.4 second sweep to produce this signal. Longer charge times for the screen increased both the signal and the background output so that no improvement in signal-to-noise ratio was obtained. This sensitivity was 1/10 of that needed to detect the highest intensity radiation expected in observing spectrums.

The vidicon was mounted in the Seemann-Bohlin camera arrangement and at attempt was made to detect the 101 plane from a quartz sample. General radiation from the sample made the background on the vidicon so high that it was difficult to find the spectrum line. With 25 KW and 16 ma of input to the x-ray tube, the 101 plane produced a signal on the oscilloscope with about a two to one signal-to-noise ratio. The vidicon appeared to be sensitive to the shorter x-ray wavelengths contained in the general radiation. The material used on the screen is proprietary

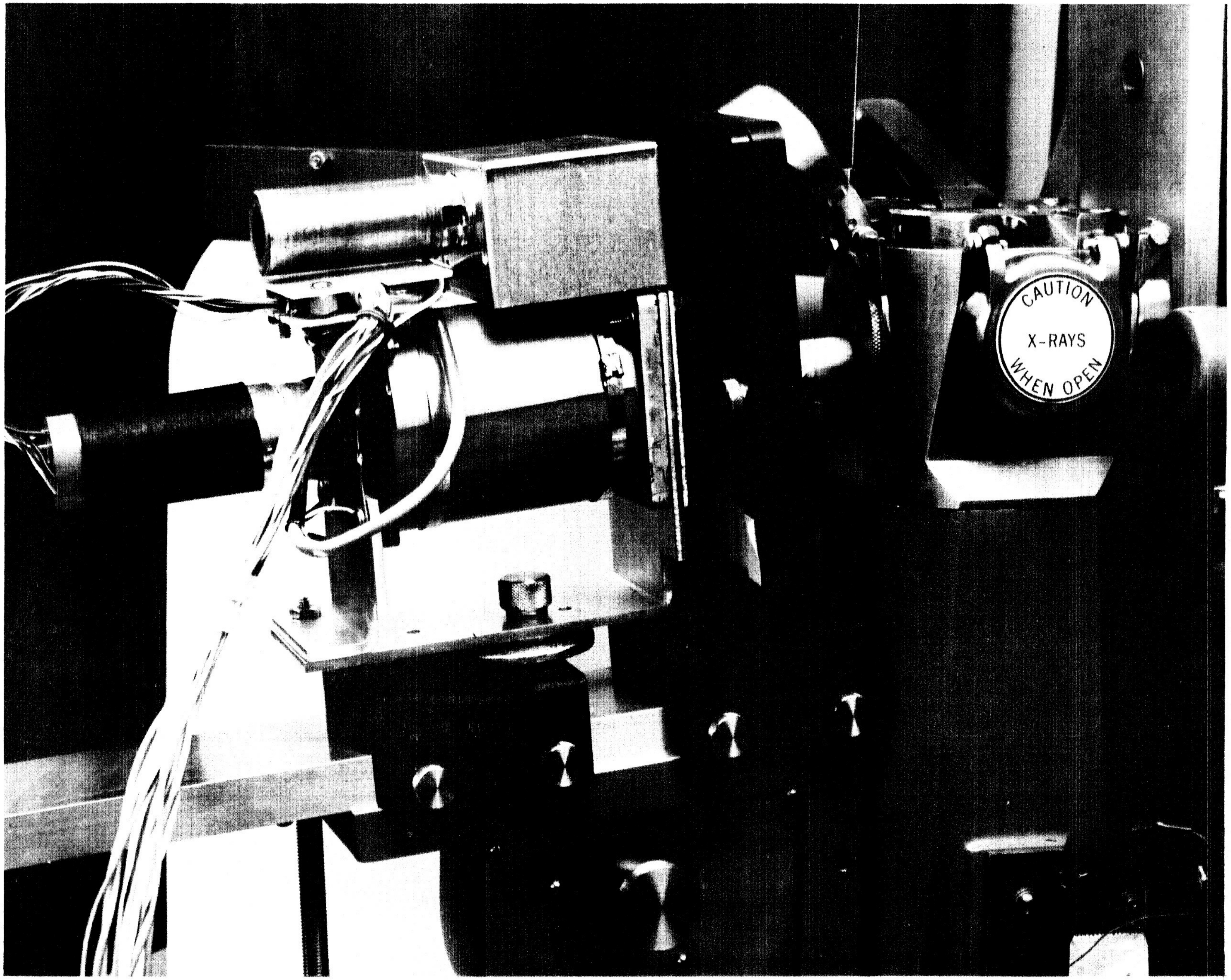


Figure 8. Vidicon Camera Assembly with Direct X-Ray Beam.

information with the manufacturer but it is known that selenium is used on x-ray sensitive vidicons. Selenium would not provide much discrimination between general radiation of wavelengths of 0.5A and characteristic radiation at wavelengths of 1.5A because the ratio of the mass absorption coefficients is only about two to one for these two wavelengths.

A special x-ray vidicon was received from the Machlett Laboratories, Inc. This tube was different in that it has no glass between the beryllium window and the photoconductive screen. Also, one-half of the screen was 30 microns thick and the other half was 15 microns thick for the purpose of evaluating the effects of screen thickness on sensitivity.

It was decided to give the new vidicon only a rough sensitivity test to determine whether it had enough increase in sensitivity to bring it near the required range. It would need to be 1000 times as sensitive as the first vidicon to be able to detect the expected weakest spectrum lines.

The tube was set up in a direct x-ray beam. When all conditions were the same as with the old tube, it was found to be twice as sensitive as the old tube. However, screen noise was not as high on the new tube, allowing more storage time, and it was possible to see 60,000 counts/sec/mm^2 with a signal-to-noise ratio of eight to one when the screen was allowed to store for two minutes. The reduced screen noise from this tube made stray noise pick-up by the high impedance target circuit more objectional so a low impedance transistor amplifier using all the target current as its base current was put in the signal amplifier.

This improved the noise problem and it was now possible to detect 15,000 counts/sec/mm^2 with a signal-to-noise ratio of three to one. These measurements were made on the 15-micron thick screen which has about twice the sensitivity of the 30-micron screen.

The vidicon sensitivity of 15,000 counts/sec/mm^2 was still so poor, as compared to the necessary sensitivity needed to see the weakest expected signal of 100 counts/sec/mm^2, that no additional work was done with it.

B. GEIGER TUBES

Two parallel plate geiger tubes were made by LND Inc. The first tube did not have a satisfactory plateau curve. The second tube, shown in Figure 9, had parallel plate electrodes separated by 0.062" with a mica window at one end and filled with neon gas at one-half atmosphere of pressure. The plateau curve for this tube is shown in Figure 10. The unamplified output pulse across the load resistor was 13 volts at the center of the plateau (500V). This output was developed by a geiger tube current of 13 microamperes. This tube was mounted in the Seemann-Bohlin camera and used to obtain an x-ray diffraction pattern (shown in Figure 11) from a powdered quartz sample. With this detector the 101 reflection had an intensity of 170 c/s, a 10 to one peak-to-background ratio, and resolution at half height of 0.6° 2θ.

In an x-ray diffractometer these parallel plate geiger tubes would be closely spaced in staggered rows around the useful part of the

Figure 9. Parallel Plate Geiger Tube

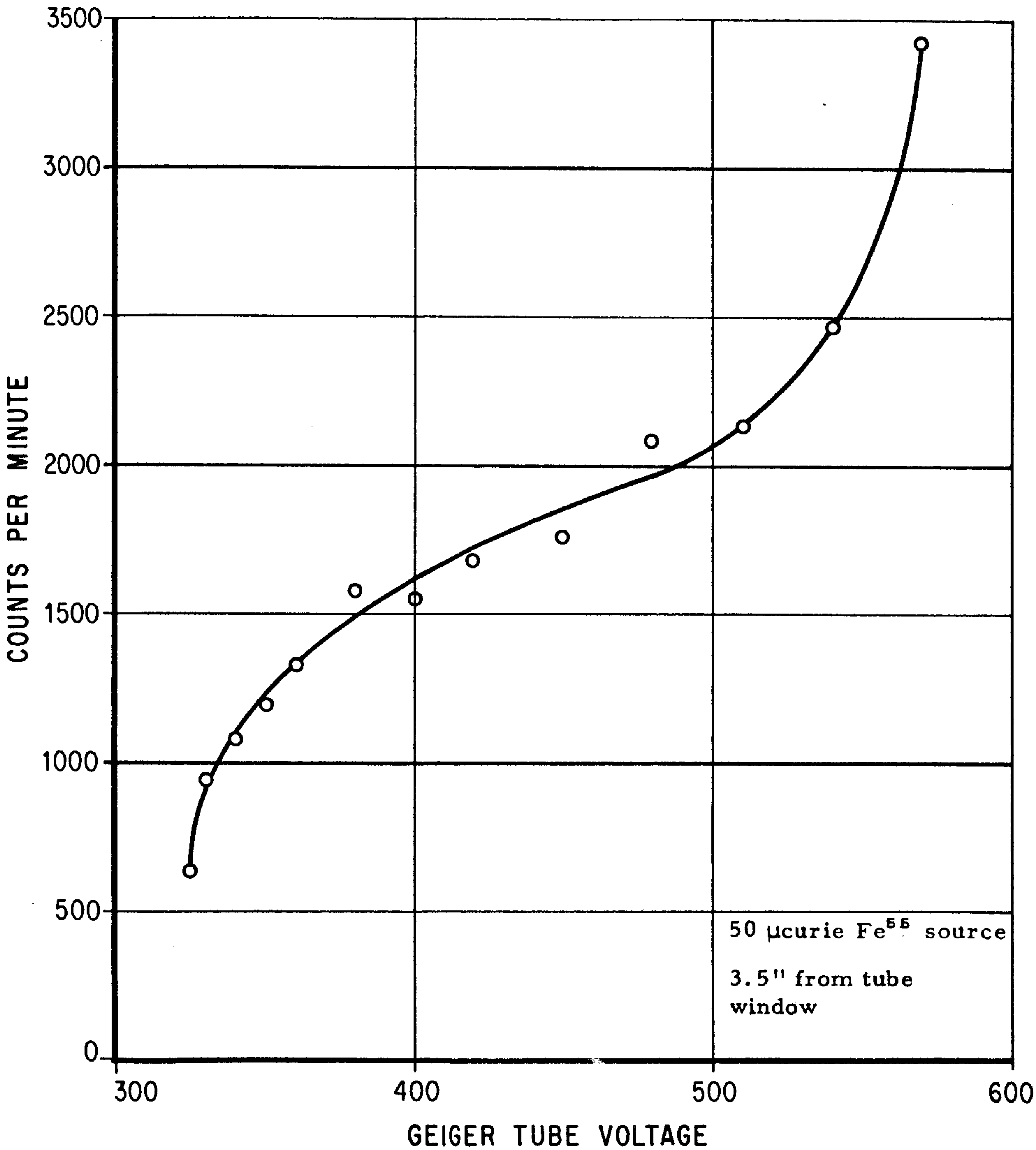


Figure 9. Plateau Curve for LND Inc. Geiger Tube.

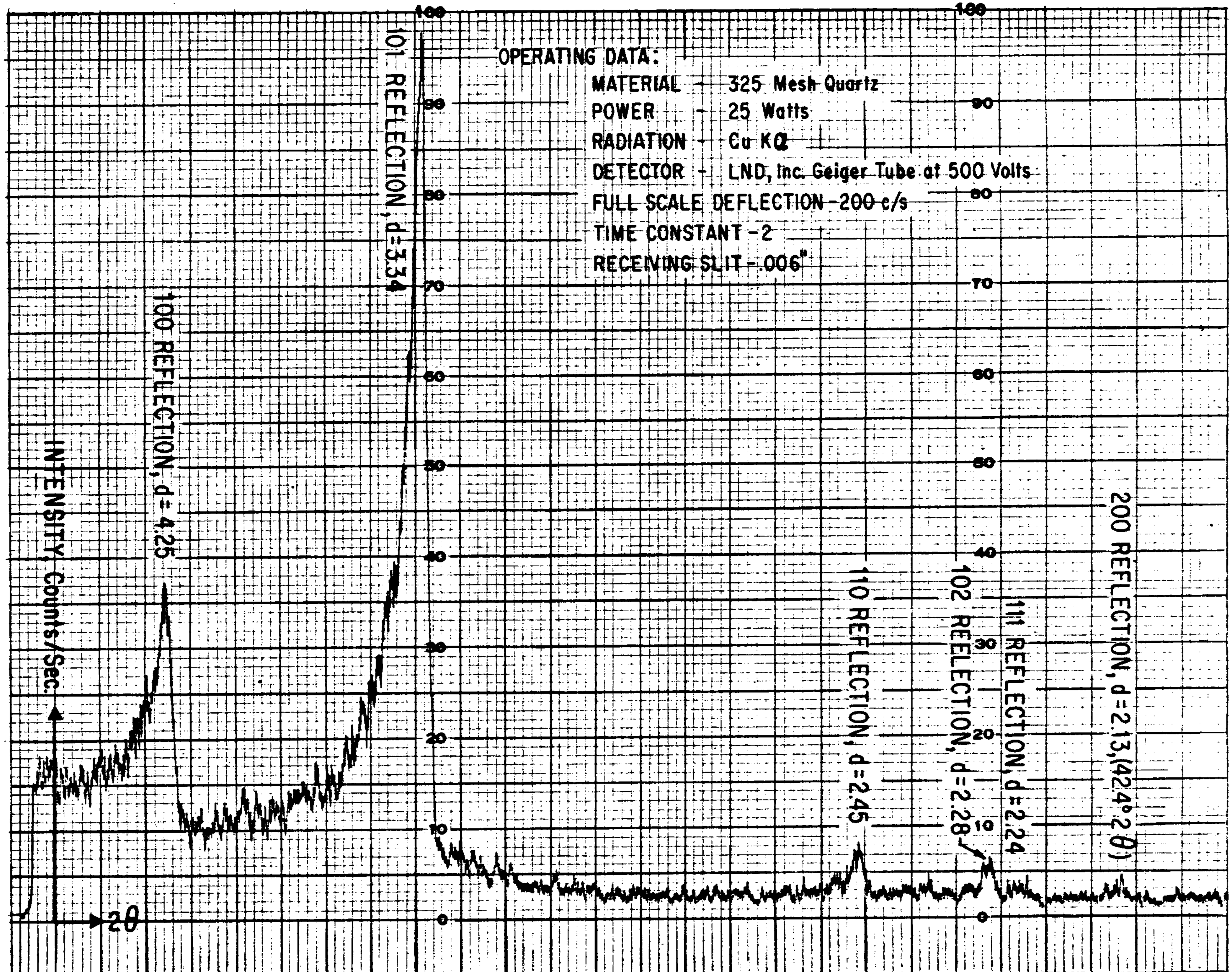


Figure 11 X-Ray Diffraction Pattern Using an LND Inc. Geiger Tube and Focusing Optics.

Rowland circle of a Seemann-Bohlin camera. Figure 12 shows a multiple flow-geiger tube constructed at the Alexandria Division of AMF. This tube was constructed as shown in Figure 13 using two end plates with five chamber insulators and four chamber plates sandwiched together and held with machine bolts and nuts. The fine wire was held to the epoxy glass insulating board with epoxy cement after assembly, a 1/4 mil thick mylar window was cemented over the open ends of the chambers. As can be seen in the drawing, gas ports were provided between chambers to provide a lengthwise flow of gas through all ten chambers in series. Electrical connections were made to the tabs on the chamber plates and the wires extending from the rear of the chambers.

When a gas of 90 percent argon and 10 percent methane was passed through the tube a plateau of 75 volts was obtained at about 600 volts. The pulse output across the tube load resistors was about 20 volts and the peak tube current was 20 microamperes. The quantum count efficiency of the tube was 45 percent and the dead time was about 50 microseconds.

When all ten channels of the tube were made to operate, some cross-talk between channels due to pick-up by the high impedance electrical circuits was noticed. However, the ratio of signal-to-crosstalk amplitude was large enough to make crosstalk no problem.

Of the two types of detectors evaluated, the geiger tube meets the detector requirements desired best. It has been shown that it is capable of producing a spectrum from a material sample, that it has high output amplitude, and that a closely spaced multiple tube can be built. The

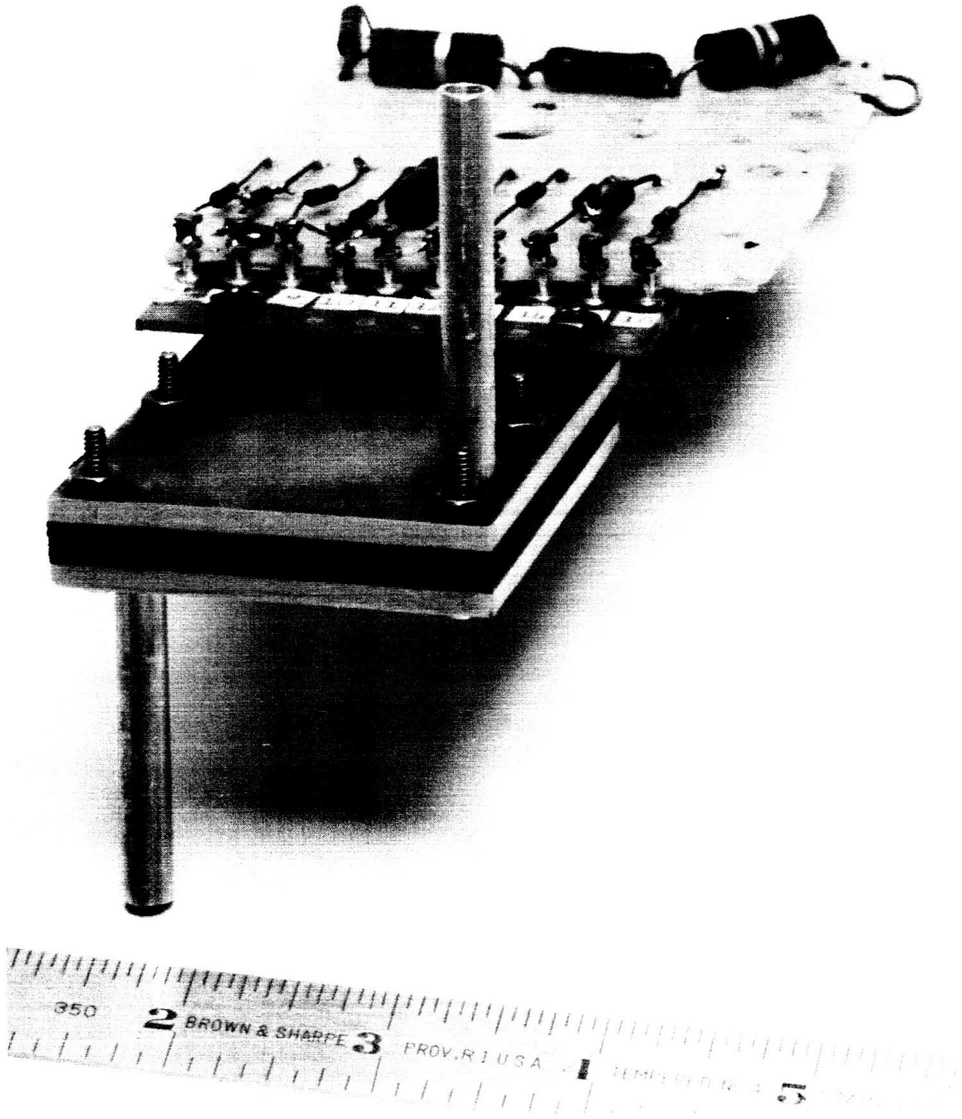


Figure 12. Multiple Flow-Geiger Tube

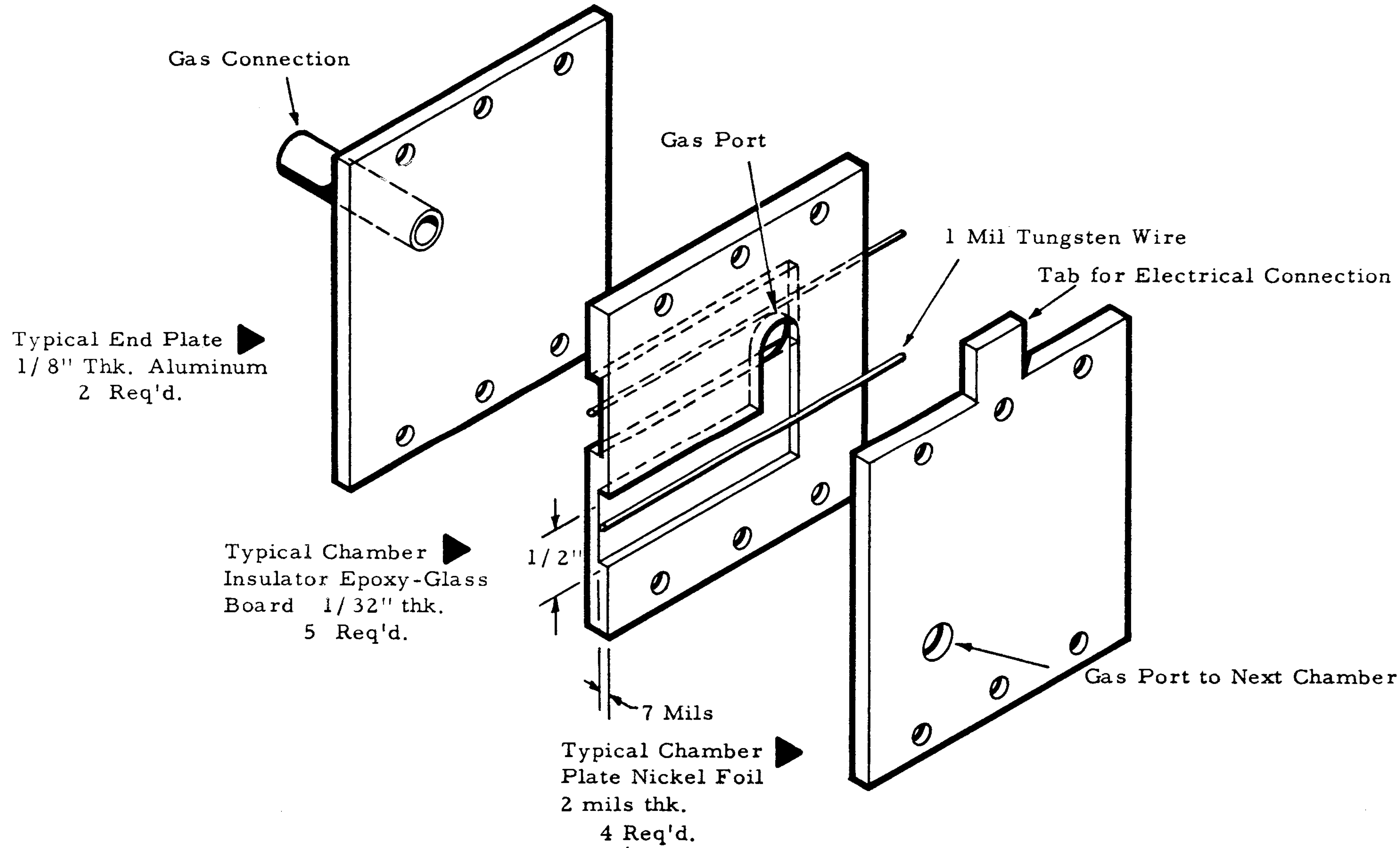


Figure 13. Construction of Multiple Flow-Geiger Tube.

problem of building a multiple chamber tube curved to match the Rowland circle still remains. This tube would need each section made to the proper thickness to give the desired resolution. For a constant resolution the thickness of the geiger tube sections must decrease as the Bragg angle decreases, making construction of a multiple geiger tube difficult.

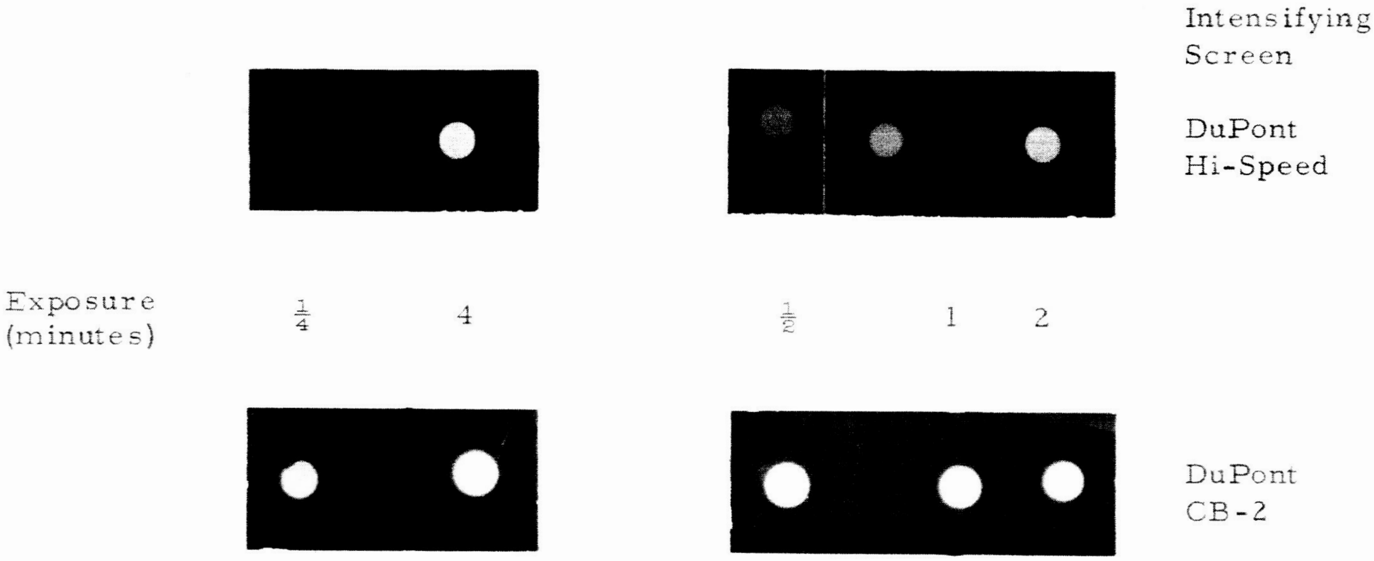
The vidicon tube is capable of resolutions much higher than is required and its resolution is nearly independent of the Bragg angle. Sensitivity is the main problem with the vidicon. One possible solution would be an intensifier on the vidicon. This solution has the disadvantage of requiring approximately 15 KV of high voltage for operating the intensifier.

C. FILM

The advent of high-speed Polaroid film with a ten-second development time and the compatability of film to a cylindrically shaped focusing camera makes this method of radiation detection worthy of consideration. Two Polaroid films are available which have merit for this application: Type 57 (ASA equivalent exposure index - 3200) and an even faster industrial film, type 410 (ASA: 10,000). All experimental work was done with type 57 film using 4" x 5" film packets. All evidence indicates that data obtained with the slower film would be proportional to results obtained with faster film.

The use of a fluorescent screen will reduce exposure time or, conversely, for the same time cause a more intense diffraction line on the film. For best results the active side of the fluorescent screen should lie adjacent to the emulsion on one side only as opposed to an acetate negative with emulsion on both sides. Hence, for maximum gain the screen must be inserted into the packet in the dark room and exposure made by passing the beam through the negative. Two commercially available fluorescent screens were used: DuPont "Hi-Speed" screen and DuPont "CB-2" screen. The former is a barium-lead sulfide intensifying screen and is moderately fast with good resolution while the latter is a zinc-cadmium sulfide fluorscopic screen which is very fast but with poorer resolution.

An example of the achievable variations of time and intensity obtainable with these screens and Polaroid film is shown in Figure 14. This experiment was conducted by allowing direct radiation to pass through a cylindrical tube, 2" x 0.187" i.d., which to an extent acted as a collimator. The tube was positioned so that one end was as close as possible to the x-ray tube (CuKα radiation) while the film packet was firmly pressed against the opposite end. Exposure times, starting with 1/4 minute, were increased by a factor of two until a maximum of four minutes was reached. The difference in intensity for varying exposure times was found to be much greater with the "Hi-Speed" screen than with the "CB-2" screen. A 1/4 minute exposure to radiation with an intensity of 2700 c/s/mm^2 using the "Hi-Speed" screen produced an image that was barely discernable; whereas an image produced with the "CB-2" screen at the same exposure



Detection of X-radiation with Polaroid Film.

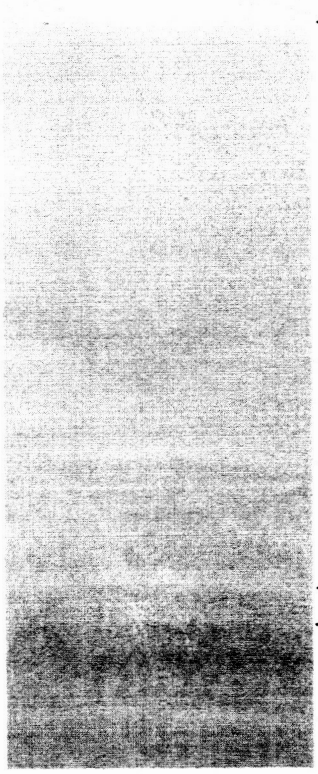
Operating Data:

Power - 25 watts - direct beam
Intensity - 2700 c/s per mm^2
Polaroid Film - type 57
(ASA exposure index - 3200)
Intensifying Screens - as shown above

Figure 14. Effect of Time on Intensity for Polaroid Film Exposed to X-Radiation

time was much stronger and would probably be of suitable intensity for most diffraction work. Furthermore, it will be noted that with the "CB-2" screen the intensity of the 1/2-minute exposure almost equals the intensity of the four-minute exposure. The "CB-2" screen is much faster because of its larger grain size and different phosphor. Consequently, poorer resolution is obtained as illustrated by the fuzziness of interface between the white dot and the black background.

X-ray diffraction patterns were taken of powdered alpha quartz using Polaroid film and a "Hi-Speed" intensifier screen. A film packet holder was first constructed which would hold the packet in a curved position equal to that of the Rowland circle curvature. By mounting the holder on the modified Seemann-Bohlin camera, patterns such as shown in Figure 15 and 16 were readily obtained in six minutes. Inasmuch as this film covers only a small segment of the 2θ spectrum, the 101 reflection was chosen as the reference line. In Figure 15 the linear distance between each of the diffracted lines (i.e. 101 to the 110; 110 to the 102, etc.) is in correct proportion to the ASTM reference data. Also, visual estimates of the intensity of the diffracted lines agree with reference data. Signal-to-background ratio is estimated to be on the order of ten to one for the 101 reflection. This is in general agreement with the results of other detectors which do not use discrimination of some type.



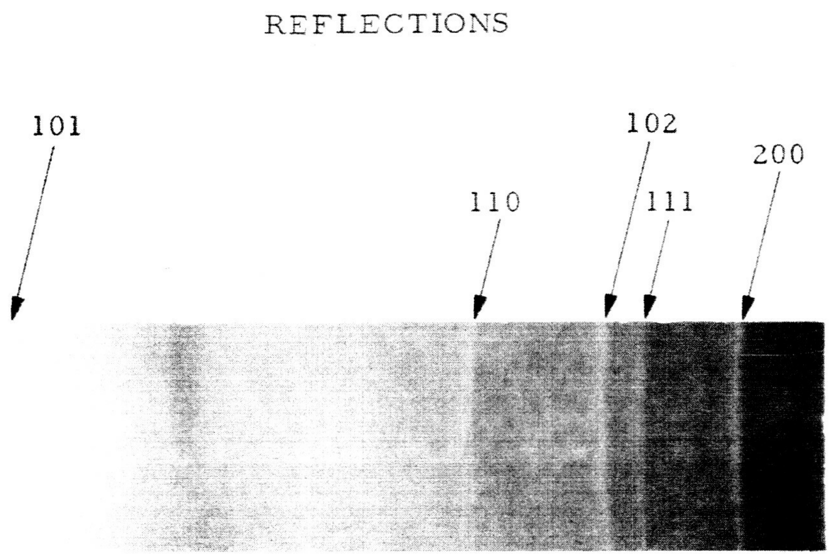
ASTM REFERENCE No. 5-0490

Reflection Plane (hkl)	Interplanar Spacing A	Relative Intensity
101	3.34	100
110	2.46	12
102	2.28	12
111	2.24	6
200	2.13	9

OPERATING DATA: CuKα radiation
Power - 25 watts
Polaroid Film - type 57
(ASA Exposure Index - 3200)
Intensifying Screen - DuPont Hi-Speed
Exposure Time - 6 minutes

Note: Original Photograph Showed Substantially Higher Contrast which was Lost in Reproduction.

Figure 15 X-Ray Diffraction Pattern of α Quartz Using Polaroid Film.



Operating Data:

Power - 25 watts - CuKα
Polaroid Film - type 57
(ASA exposure index - 3200)
Intensifying Screen - DuPont CB-2
Exposure Time - 6 minutes
Forward Reflection Region -
101 (26.7° 2θ) to 200 (42.4° 2θ)

Figure 16. X-Ray Diffraction Pattern of Alpha Quartz Using Polaroid Film

Summarizing the required number of photons per second per square milimeter is in the order of 6×10^7 for DuPont "Hi-Speed" intensifier screen and about 4×10^6 for DuPont CB-2 screen corresponding to a density of about 0.8. The sensitivity of these films without the intensifier screen is down by a factor of 10 to 20. Thus, the use of Polaroid film in combination with fluorescent screens offers a system of x-radiation detection which can quickly, easily, and simultaneously record a diffraction pattern. Potential technical areas of study for improving the signal-to-background ratio and the resolution appear to be: (1) a better matching of the film sensitivity to a particular wavelength of incident x-radiation; (2) film sensitivity to the particular wavelength of the light emitted by the excited phosphor; (3) an evaluation of several films with differing characteristics, including grain size and the compatability of each film to particular fluorescent screen; (4) an improvement in the mechanical arrangement to ensure uniform and intimate contact of the screen and the negative; and (5) an investigation of filters or other devices such as a crystal monochromator to reduce bremsstrahlung or hard radiation scatter and thus enhance the signal-to-background ratio. Cursory experiments indicate that Polaroid film type 57 probably has sufficient inherent resolution for x-ray diffraction purposes, if the intensity is sufficient.

IV. DATA HANDLING AND PRESENTATION

The data handling and presentation system fully reproduces all functions which are to be performed in space, but only ten channels of primary information are handled by the feasibility model. The system concept calls for storing the outputs of ten channels and electronically scanning the ten channels along with a sync signal and transmitting this information in serial fashion on a single line. The serial information is then selectively gated into individual channels and fed into a six-digit counter which is switched manually between channels. It was decided, for the feasibility model, to utilize commercially available logic circuitry to the fullest extent possible. This was decided primarily for economic reasons because this is a feasibility study and size at this point is not a factor. The feasibility model is housed in a desk-top 19-inch rack which contains all data reduction circuits and the controls for readout. The feasibility model card rack and control panel are shown in Figure 17.

The system was designed to be applicable to any detector producing output counts or pulses, but an array of amplifiers was designed specifically to match the output of a geiger tube. A block diagram of the system logic is shown in Figure 18. Channels 3 through 9 are omitted from the drawing, but all ten channels have been constructed.

The data handling system consists primarily of a system clock, scanning circuits, and ten information channels plus a sync signal. Incoming information from the ten individual channels of the geiger tube

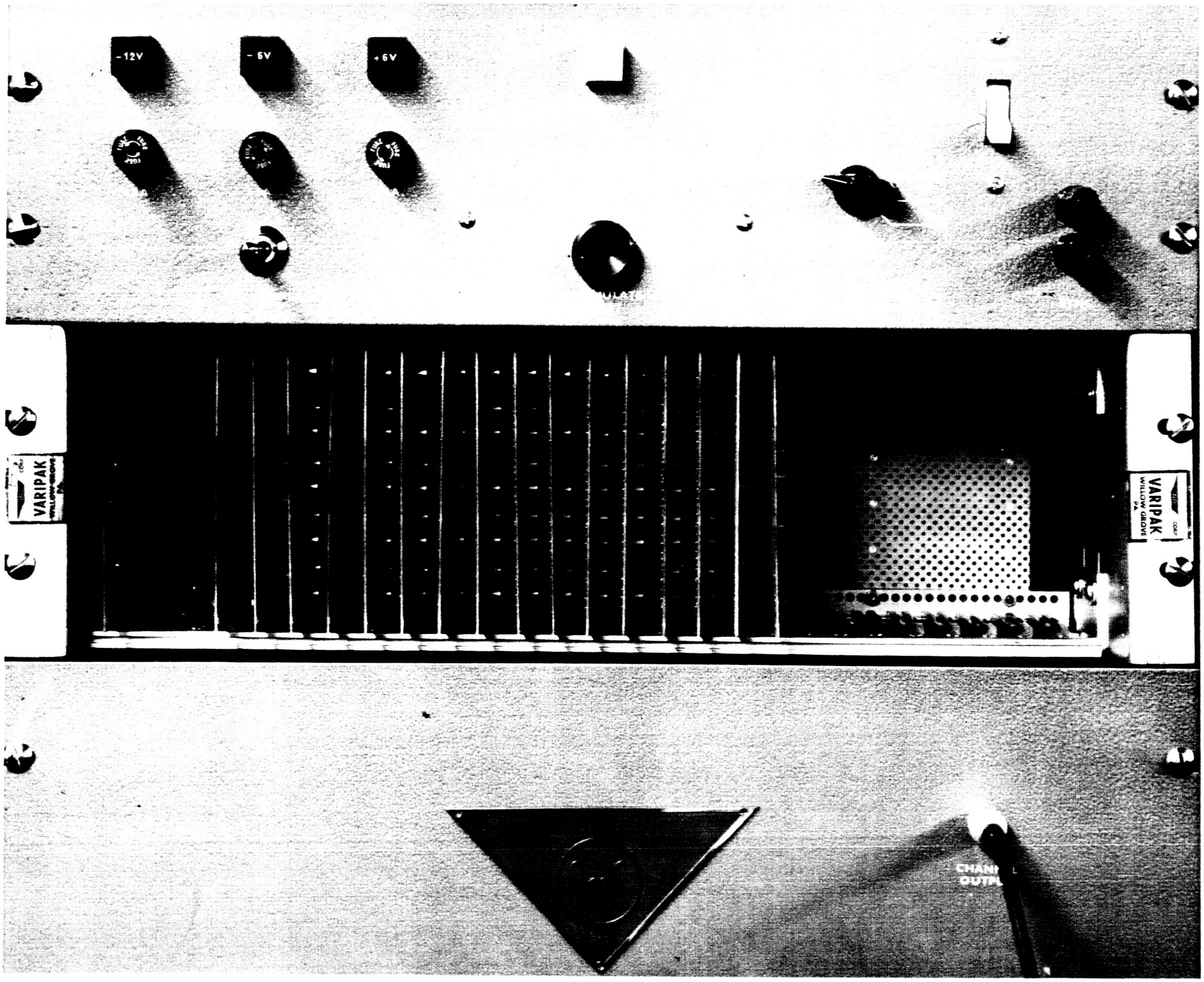


Figure 17 Data Reduction Circuits and Readout Controls.

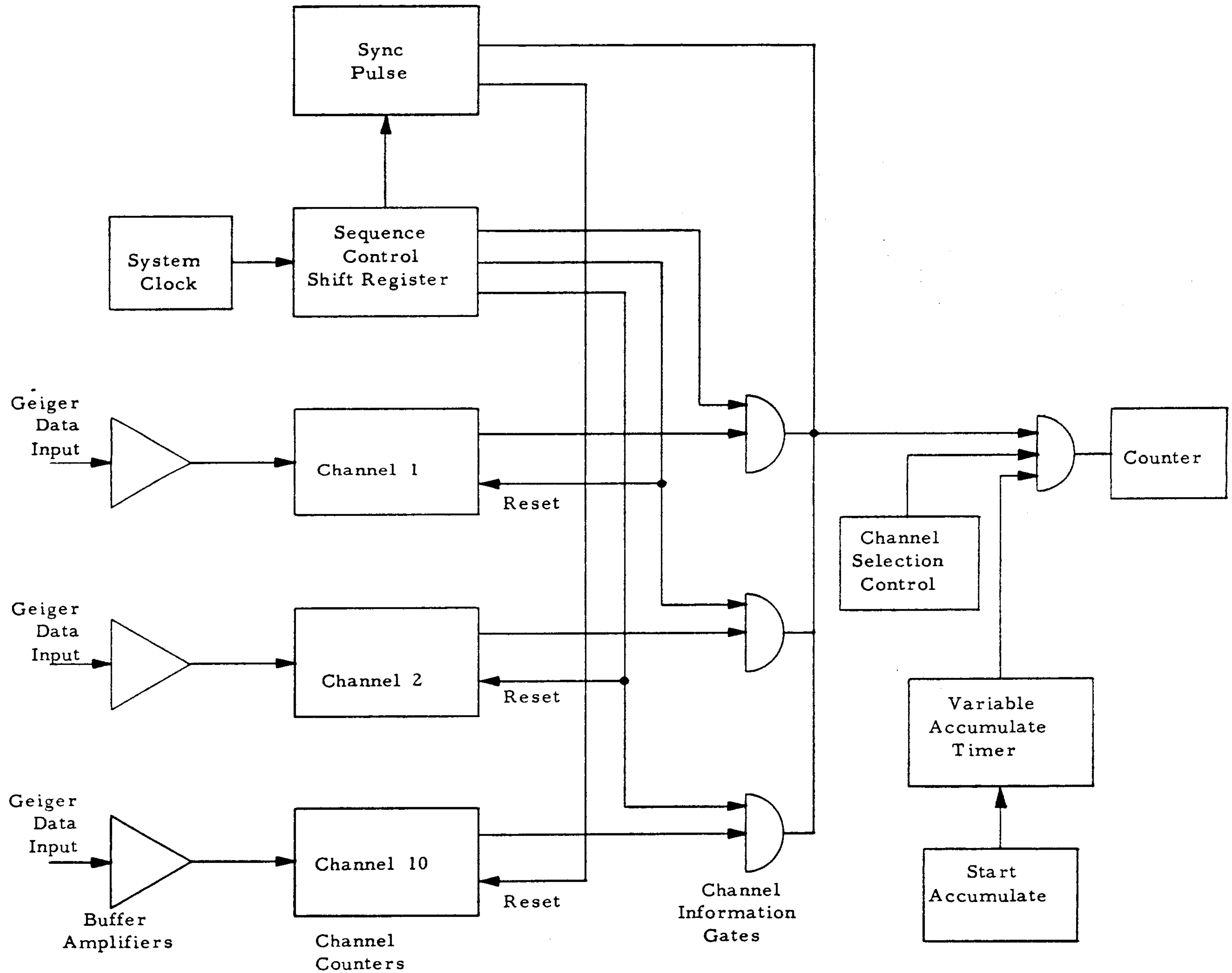


Figure 18. Abbreviated Block Diagram of Data Accumulation and Display System

are fed into N/4 counters to provide temporary storage until the output of the channels is controlled by the clock rate of 30 kc. This rate allows for simultaneous inputs on each of the ten channels varying independently from zero to more than 10,000 counts per second. The present data handling system could handle an incoming data rate of up to 36,000 counts per second per channel simply by increasing the clock frequency to its maximum rate of 100 kc. Commercial logic cards of the type shown in Figure 19 were used throughout the system with the exception of the input buffer amplifiers. Special input amplifiers were designed to make the transition between the geiger tube outputs and the input requirements of the logic circuits. As the outputs of the channels are scanned, the information is gated onto a single line in serial fashion for transmission to the readout point. In a flight instrument the readout point would be the spacecraft telemetry system.

After the data processing and presentation equipment were built, it was connected with its supporting equipment for a system check. All data reduction channels and the controls were checked and found to perform well within design specifications.

Selective readout was chosen rather than simultaneous readout primarily to reduce the cost of the feasibility model. After choosing the channel with the selector switch on the master control panel, the "Accumulate" button is depressed momentarily to activate the readout circuits for a predetermined period of time. The count occurring during this

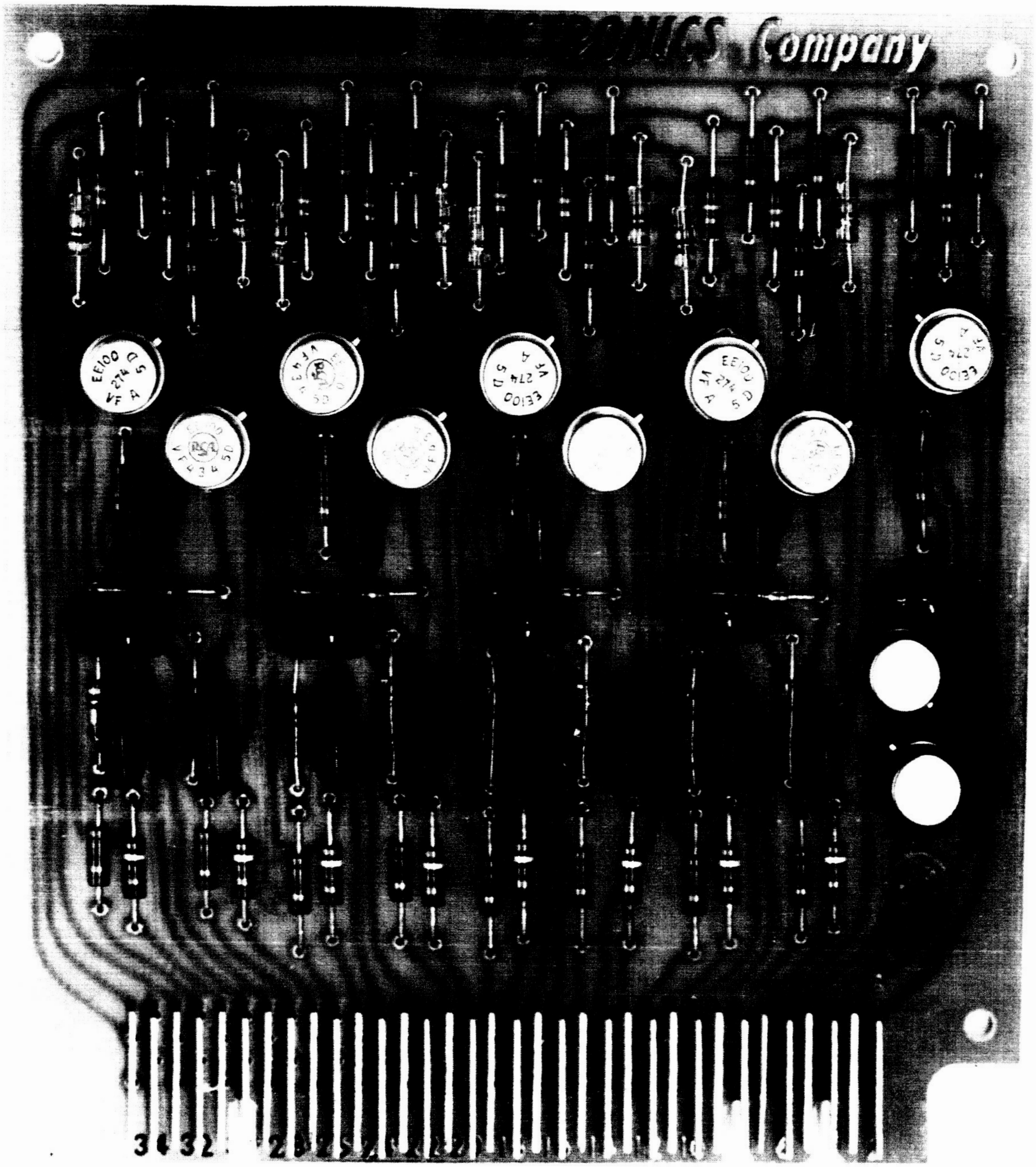


Figure 19 Commercial Logic Card Used in Data Reduction Circuits.

time is then accumulated and displayed on a Hewlett-Packard counter. Pressing the "Accumulate" button activates an audio oscillator and a C.M.C. preset counter which are used to control the length of time that data is presented to the counter. Time intervals for accumulation may be varied from one second to 100 seconds with an accuracy greater than 1 percent. Varying the time for data accumulation is controlled by changing the oscillator frequency and/or the preset count.

The techniques used in the feasibility model to put all the information on one line and recover individual channel information are directly applicable to a 500-channel system. In the larger system the clock rate would have to be increased to approximately 1.25 megacycles or the capacity of the channel counters would have to be increased to gain sufficient temporary storage. An oscillator and shift register duplicating the ones in the transmitting unit would be needed at the receiving end to recover the individual channel information. By detecting the sync signal in the information stream and using it to synchronize the oscillator and the shift register, recovery of the information becomes exactly the reverse of the process used to put it into serial form. The ten-channel feasibility system utilizes the same oscillator and shift register for reducing the data to serial form and recovering it.

As a result of this effort it is clear that the detector outputs can be handled in a manner economical in terms of size, weight, cost, and telemetry capacity. The performance with a particular detector, such as the multiple geiger tube, remains to be demonstrated but it is not expected to present any unusual problems.

V. CONCLUSIONS AND RECOMMENDATIONS

The research program described in this report pertains to the study of detector systems and compatible optic arrangements suitable for simultaneous detecting and recording an x-ray diffraction spectrum without the use of mechanical scanning devices. This program was conducted to establish feasibility and design concepts utilizing multiple or stacked detectors, a single fixed detector, or other pertinent x-ray detecting systems. The experimental program was approached from three viewpoints: x-ray optics, detecting systems, and data handling and presentation.

The most advantageous optical design was found to be the focusing geometrical arrangement similar to that found in Seemann-Bohlin cameras. The use of an asymmetrical arrangement is preferred as opposed to symmetrical geometry because it permits lower Bragg angle reflections to be obtained. A prime advantage of the asymmetrical focusing optical design for analytical x-ray diffraction analysis is due to the fact that cameras can be constructed which give much greater resolution and intensity without increased exposure time as compared with other optical arrangements. On the average, curved optics will increase the intensity of an x-ray beam by a factor of 16 to one when compared to a flat optic system. Furthermore, specimen preparation, loading, and alignment are less complex.

Two prototype focusing cameras were designed and constructed. In order to show design feasibility, the optical system was evaluated with a standard scintillation detecting system using pulse height discrimination. The detector and discriminator were originally partof a standard laboratory x-ray diffractometer. With this system, the peak intensity of the 101 reflection of powdered alpha quartz was approximately 10,000 counts per second or about twice the value obtained on the Surveyor Bragg-type diffractometer under equivalent conditions. The resolution was $0.3^{\circ}2\theta$, and the peak-to-background ratio was about fifty to one. Theoretical considerations indicate that intensity and signal-to-background ratio should improve even further if a smaller Rowland circle is used.

Two types of detectors were evaluated: the geiger tube and the x-ray vidicon. The geiger tube had the required sensitivity and good discrimination between the desired spectrum radiation and the undesired background radiation. It still needs to be proven that a multiple geiger tube of the correct configuration and with the required resolution can be built. It is recommended that a multiple chamber geiger tube be built and used as a detector in a Seemann-Bohlin camera along with the electronics to process the output.

The x-ray vidicon detector proved to have better than required resolution but fell short of the required sensitivity. The minimum expected peak intensity would be 100 counts/sec/mm^2 but the lowest detectable peak intensity obtained with the vidicon was 15,000 counts/sec/mm^2 with a signal to noise ratio of three to one. From this it can be seen that the sensitivity would need to be improved by a factor of at least 150 or even more to improve the signal-to-vidicon-noise ratio. It is recommended that an intensifier be added to the vidicon to increase the sensitivity. This intensifier would provide an increase in sensitivity of 1000 without adding any noise. However, resolution of the intensifier is poorer than the resolution of the vidicon alone and would require verification that it is good enough as a detector for this focusing camera.

The use of high-speed Polaroid film when combined with fluorescent intensifying screens also provides a suitable method of detecting and recording an x-ray diffraction spectrum. Exposure times of six minutes or less were shown to be feasible for recording a portion of an alpha quartz x-ray diffraction pattern. Recorded d values and relative intensities (visually estimated) were in agreement with ASTM reference data. Further work in this area is recommended. Primary emphasis should be given to increasing the blackening of the film for incident radiation of fixed intensity and to exploring the sensitivity range of film-intensifier screen combinations.

A data handling and presentation system was designed and constructed in order to provide economical and realistic representation of the functions of a space system. All logic fuctions were obtained with commercially available NAND-NOR logic cards, primarily because this type of logic could perform all necessary operations with a minumum number of components. The method of data handling is centered around the sequence controlling shift register and the channel input counters used for temporary storage. The feasibility model is capable of converting ten channels of input data, varying independently from zero to more than 10,000 counts per second, to an output on a single line from which individual channel information can be recovered. The readout used in recovering individual channel information utilized, wherever possible, commercial equipment in lieu of specially designed circuits.